#### **REMARKS**

For the amendments set forth herein, the applicant submits that the application is now in condition for allowance and early allowance is requested at the Examiner's earliest convenience.

Respectfully submitted,

Date:

WILDMAN, HARROLD, ALLEN & DIXON LLP

225 West Wacker Drive

Chicago, IL 60606 Ph. 312/201-2000

Fax 312/201-2555

By:\_

Douglas S. Ruper

Reg. No. 44,434

CERTIFICATE OF EXPRESS MAIL

"Express Mail" mailing label number: 1986895229
Date of Deposit: 9-9-03

I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" on the date indicated above and is addressed to the Commissioner for Parents, P.O. Box 1450 Alexandria, VA

22313:1450. Wen a TUCK

# IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of Harry W. Sarkas et al.	) PROCESS FOR PREPARING
21.10 upp	) NANOSTRUCTURED MATERIALS
	OF CONTROLLED SURFACE
	) CHEMISTRY
Serial No.: 10/172,848	)
,	Attorney Docket: 2000US01
Filed: June 17, 2002	)
	) Group Art Unit: N/A
<i>;</i>	)
	•

Assistant Commissioner for Patents Washington, D.C. 20231

## PRELIMINARY AMENDMENT

Dear Sir:

Applicant hereby submits the following Preliminary Amendment to the above-referenced application.

### **AMENDMENTS**

Please amend the above-identified application.

Please replace pages 10 through 15 with replacement pages 10 through 16 enclosed herewith.

### **REMARKS**

In response to Notice of Incomplete Reply dated October 9, 2002, enclosed is replacement page 10 of specification, new pages 11-15 of the claims and new page 16 of the Abstract. Replacement pages were necessary to overcome the rejection of the specification and claims being on the same page. Applicant asserts no new subject matter has been added and that these amendments are to correct formatting. A marked up version of the claims is provided to show the changes.

Respectfully submitted,

Douglas S. Rupert

Attorney for Applicants Reg. No. 44,434

Date: 10/28/0

WILDMAN, HARROLD, ALLEN & DIXON

225 West Wacker Drive

Chicago, IL 60606

Ph. 312/201-2000

Fax 312/201-2555

The preceding specific embodiments are illustrative of the practice of the invention. It is to be understood, however, that other expedients known to those skilled in the art, or disclosed herein, may be employed without departing from the spirit of the invention or the scope of the appended claims.

#### We Claim:

5

10

20

1. A process to prepare stoichiometric-nanostructured materials comprising:

generating a plasma;

forming an "active volume" through introduction of an oxidizing gas into the plasma, before the plasma is expanded into a field-free zone, either (1) in a region in close proximity to a zone of charge carrier generation, or (2) in a region of current conduction between field generating elements, including the surface of the field generation elements; and

transferring energy from the plasma to a precursor material or materials and forming in the "active volume" at least one of stoichiometric-nanostructured materials and a vapor that may be condensed to form a stoichiometric-nanostructured material.

- 2. The process of claim 1, wherein the step of generating comprises utilizing a radiofrequency field to generate the plasma.
- 3. The process of claim 1, wherein the step of generating comprises utilizing a microwave discharge to generate the plasma.
  - 4. The process of claim 1, wherein the step of generating comprises utilizing a free-burning electric arc to generate the plasma.
  - 5. The process of claim 1, wherein the step of generating comprises utilizing a transferred electric arc to generate the plasma.
  - 6. The process of claim 1, wherein the step of generating comprises utilizing a highintensity laser to generate the plasma.

- 7. The process of claim 1, wherein the step of generating comprises utilizing a capacitively coupled electro-thermal igniter to generate the plasma.
- 8. The process of claim 1, wherein the step of generating comprises utilizing a DC glow discharge to generate the plasma.
- 5 9. The process of claim 1, wherein the step of generating comprises utilizing a DC cold cathode discharge to generate the plasma.
  - 10. The process of claim 1, wherein the step of forming comprises selecting the oxidizing gas from one of a gas containing oxygen atoms or a gas mixture containing oxygen atoms.
  - 11. The process of claim 1, wherein the step of forming comprises selecting non-oxygen components of the oxidizing gas from a group comprising He, Ne, Ar, Kr, Xe, N<sub>2</sub>, and H<sub>2</sub>, or mixtures thereof.

- 12. The process of claim 1, wherein the step of forming comprises selecting  $N_2O$  as the oxidizing gas.
- 13. The process of claim 1, wherein the step of forming comprises selecting  $O_2$  as the oxidizing gas.
- 14. The process of claim 1, wherein the step of forming comprises selecting CO<sub>2</sub> as the oxidizing gas.
- 15. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas into a anodic column of a transferred electric arc.
- 20 16. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas into a cathodic column of a transferred electric arc.

- 17. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas into a anodic column of a free-burning electric arc.
- 18. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas into a cathodic column of a free-burning electric arc.
- 5 19. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas to the plasma by natural convection.
  - 20. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas to the plasma by forced convection.
  - 21. The process of claim 1, wherein the step of forming comprises allowing the oxidizing gas to atomize a liquid nanoparticle precursor and introduce it into the "active volume".

- 22. The process of claim 1, wherein the step of forming comprises allowing the oxidizing gas to fluidize and transport a solid nanoparticle precursor into the "active volume".
- 23. The process of claim 1, further comprising:

  Injecting at least one of a quench and dilution stream just beyond the "active volume."

  The injection point beyond the "active volume" is from one mean free path of a plasma species to a larger distance deemed to be appropriate to quench the vapor and is generally determined by process equipment configuration.
  - 24. The process of claim 23, wherein the step of injecting comprises creating a nanoparticle aerosol of controlled particle size.
- 25. Stoichiometric-nanostructured materials produced through steps comprising: generating a plasma;

forming an "active volume" through introduction of an oxidizing gas into the plasma, before the plasma is expanded into a field free zone, in a region in close proximity to either (1) a zone of charge carrier generation, or (2) a region of current conduction between field generating elements, including the surface of the field generating electrodes; and

transferring energy from the plasma to a precursor material or materials and forming in the "active volume" at least one of stoichiometric-nanostructured materials and a vapor that may be condensed to form a stoichiometric-nanostructured material.

26. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials are metal oxides.

5

10

15

- 27. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials are substantially spherical nanocrystalline metal oxides.
- 28. The stoichiometric-nanostructured materials of claims 26 and 27, wherein the metal oxides are selected from a group comprising aluminum oxide, zinc oxide, iron oxide, cerium oxide, chromium oxide, antimony tin oxide, mixed rare earth oxides, and indium tin oxide.
- 29. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials generally have a size distribution and range in mean diameter from about 1 nm to about 900 nm.

- 30. The stoichiometric-nanostructured materials of claim 29, wherein the stoichiometric-nanostructured materials generally have a size distribution and range in mean diameter from about 2 nm to about 100 nm.
- 31. The stoichiometric-nanostructured materials of claim 30, wherein the stoichiometric-nanostructured materials generally have a size distribution and range in mean diameter from about 5 nm to about 40 nm.

10

15

- 32. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials have a surface chemistry having a high aqueous dispersion stability.
- 33. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials have a surface chemistry having a low rate of hydrolysis.
  - 34. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials have a surface chemistry with the absolute value of the zeta potential greater than 20 mV.
  - 35. The stoichiometric-nanostructured materials of claim 34, wherein the stoichiometric-nanostructured materials have a surface chemistry with the absolute value of the zeta potential greater than 30 mV.
    - 36. The stoichiometric-nanostructured materials of claim 35, wherein the stoichiometric-nanostructured materials have a surface chemistry with the absolute value of the zeta potential greater than 35 mV.

### **ABSTRACT**

A process to prepare stoichiometric-nanostructured materials comprising generating a plasma, forming an "active volume" through introduction of an oxidizing gas into the plasma, before the plasma is expanded into a field-free zone, either (1) in a region in close proximity to a zone of charge carrier generation, or (2) in a region of current conduction between field generating elements, including the surface of the field generation elements, and transferring energy from the plasma to a precursor material to form in the "active volume" at least one stoichiometric-nanostructured material and a vapor that may be condensed to form a stoichiometric-nanostructured material. The surface chemistry of the resulting nanostructured materials is substantially enhanced to yield dispersion stable materials with large zeta-potentials.

10

The preceding specific embodiments are illustrative of the practice of the invention. It is to be understood, however, that other expedients known to those skilled in the art, or disclosed herein, may be employed without departing from the spirit of the invention or the scope of the appended claims.

Page break

### We Claim:

5

10

20

1. A process to prepare stoichiometric-nanostructured materials comprising:

generating a plasma;

forming an "active volume" through introduction of an oxidizing gas into the plasma, before the plasma is expanded into a field-free zone, either (1) in a region in close proximity to a zone of charge carrier generation, or (2) in a region of current conduction between field generating elements, including the surface of the field generation elements; and

transferring energy from the plasma to a precursor material or materials and forming in the "active volume" at least one of stoichiometric-nanostructured materials and a vapor that may be condensed to form a stoichiometric-nanostructured material.

- 2. The process of claim 1, wherein the step of generating comprises utilizing a radio-frequency field to generate the plasma.
- The process of claim 1, wherein the step of generating comprises utilizing a microwave discharge to generate the plasma.
  - 4. The process of claim 1, wherein the step of generating comprises utilizing a free-burning electric arc to generate the plasma.
  - 5. The process of claim 1, wherein the step of generating comprises utilizing a transferred electric arc to generate the plasma.
  - 6. The process of claim 1, wherein the step of generating comprises utilizing a highintensity laser to generate the plasma.

- 7. The process of claim 1, wherein the step of generating comprises utilizing a capacitively coupled electro-thermal igniter to generate the plasma.
- 8. The process of claim 1, wherein the step of generating comprises utilizing a DC glow discharge to generate the plasma.
- 5 9. The process of claim 1, wherein the step of generating comprises utilizing a DC cold cathode discharge to generate the plasma.
  - 10. The process of claim 1, wherein the step of forming comprises selecting the oxidizing gas from one of a gas containing oxygen atoms or a gas mixture containing oxygen atoms.
  - 11. The process of claim 1, wherein the step of forming comprises selecting non-oxygen components of the oxidizing gas from a group comprising He, Ne, Ar, Kr, Xe, N<sub>2</sub>, and H<sub>2</sub>, or mixtures thereof.

- 12. The process of claim 1, wherein the step of forming comprises selecting N<sub>2</sub>O as the oxidizing gas.
- 13. The process of claim 1, wherein the step of forming comprises selecting  $O_2$  as the oxidizing gas.
- 14. The process of claim 1, wherein the step of forming comprises selecting CO<sub>2</sub> as the oxidizing gas.
- 15. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas into a anodic column of a transferred electric arc.
- 20 16. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas into a cathodic column of a transferred electric arc.

- 17. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas into a anodic column of a free-burning electric arc.
- 18. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas into a cathodic column of a free-burning electric arc.
- 5 19. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas to the plasma by natural convection.
  - 20. The process of claim 1, wherein the step of forming comprises introducing the oxidizing gas to the plasma by forced convection.
  - 21. The process of claim 1, wherein the step of forming comprises allowing the oxidizing gas to atomize a liquid nanoparticle precursor and introduce it into the "active volume".

15

- 22. The process of claim 1, wherein the step of forming comprises allowing the oxidizing gas to fluidize and transport a solid nanoparticle precursor into the "active volume".
- 23. The process of claim 1, further comprising:

  Injecting at least one of a quench and dilution stream just beyond the "active volume."

  The injection point beyond the "active volume" is from one mean free path of a plasma species to a larger distance deemed to be appropriate to quench the vapor and is
- 24. The process of claim 23, wherein the step of injecting comprises creating a nanoparticle aerosol of controlled particle size.
- 25. Stoichiometric-nanostructured materials produced through steps comprising:
  generating a plasma;

generally determined by process equipment configuration.

forming an "active volume" through introduction of an oxidizing gas into the plasma, before the plasma is expanded into a field free zone, in a region in close proximity to either (1) a zone of charge carrier generation, or (2) a region of current conduction between field generating elements, including the surface of the field generating electrodes; and

transferring energy from the plasma to a precursor material or materials and forming in the "active volume" at least one of stoichiometric-nanostructured materials and a vapor that may be condensed to form a stoichiometric-nanostructured material.

26. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials are metal oxides.

5

10

15

- 27. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials are substantially spherical nanocrystalline metal oxides.
- 28. The stoichiometric-nanostructured materials of claims 26 and 27, wherein the metal oxides are selected from a group comprising aluminum oxide, zinc oxide, iron oxide, cerium oxide, chromium oxide, antimony tin oxide, mixed rare earth oxides, and indium tin oxide.
- 29. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials generally have a size distribution and range in mean diameter from about 1 nm to about 900 nm.

- 30. The stoichiometric-nanostructured materials of claim 29, wherein the stoichiometric-nanostructured materials generally have a size distribution and range in mean diameter from about 2 nm to about 100 nm.
- 31. The stoichiometric-nanostructured materials of claim 30, wherein the stoichiometric-nanostructured materials generally have a size distribution and range in mean diameter from about 5 nm to about 40 nm.

- 32. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials have a surface chemistry having a high aqueous dispersion stability.
- 33. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials have a surface chemistry having a low rate of hydrolysis.
  - 34. The stoichiometric-nanostructured materials of claim 25, wherein the stoichiometric-nanostructured materials have a surface chemistry with the absolute value of the zeta potential greater than 20 mV.
- 35. The stoichiometric-nanostructured materials of claim 34, wherein the stoichiometric-nanostructured materials have a surface chemistry with the absolute value of the zeta potential greater than 30 mV.
  - 36. The stoichiometric-nanostructured materials of claim 35, wherein the stoichiometric-nanostructured materials have a surface chemistry with the absolute value of the zeta potential greater than 35 mV.

## **ABSTRACT**

A process to prepare stoichiometric-nanostructured materials comprising generating a plasma, forming an "active volume" through introduction of an oxidizing gas into the plasma, before the plasma is expanded into a field-free zone, either (1) in a region in close proximity to a zone of charge carrier generation, or (2) in a region of current conduction between field generating elements, including the surface of the field generation elements, and transferring energy from the plasma to a precursor material to form in the "active volume" at least one stoichiometric-nanostructured material and a vapor that may be condensed to form a stoichiometric-nanostructured material. The surface chemistry of the resulting nanostructured materials is substantially enhanced to yield dispersion stable materials with large zeta-potentials.

10